EXPERIMENTAL MANUFACTURE OF SOAP WITH RECOVERY OF GLYCERINE

BY

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A THESIS

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THE DEGREE OF BACHELOR OF SCIENCE

IN

CHEMICAL ENGINEERING

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SUBMITTED BY

C. H. TEESDALE & ARNOLD PACYNA

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JUNE 1908.

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AN INVESTIGATION

ON

"THE EXPERIMENTAL MANUFACTURE
OF SOAP WITH RECOVERY OF
GLYCERINE."

THE EXPERIMENTAL MANUFACTURE OF SOAP WITH RECOVERY OF GLYCERINE.

Soap is the metallic salt of non-volatile fatty acids. Commercial washing soap is a mixture of various fatty acid bases with either sodiums or potassiums as the metal. The fatty stock is derived from either animal or vegetable sources, and is usually in the form of glycerides of the fatty acids. Soap is made by decomposition of the glyceride by sodium or potassium, hydroxide, with separations and purification of the resultant soap. This takes place according to the following equation.

$$C_3H_5(C_{18}H_{35}O_2)_3 + 3NaOH$$

$$C_3H_5(OH)_3 + 3C_{18}H_{35}O_2 \cdot Na$$

In the soap factory, ordinary laundry or toilet soap is made by the boiled process. This is done in several stages.

The melted fat and lye at 15° Be' are run into the kettle together, while free steam is blown in to mix them, and to form an emulsion of the oil and lye, which is essential to the beginning of the saponification. It is very essential that this emulsion be formed, or the resultant saponification will not take place properly. Strong lye is then added, a small portion at a time, and boiling is continued to complete the saponification. When completely saponified, it is grained or salted out by adding common salt. This causes a separating of the soap from lye and glycerine. The steam is cut off, and the soap allowed to stand for several hours, when it rises to the top and solidifies. The salt lye and glycerine is drawn off, and treated for the recovery of glycerin.

Strong lye is now added, and for yellow soaps rosin is introduced; for white soaps, tallow or cocoanut oil are used instead of the rosin. The boiling is continued for some hours until the soap becomes clear and semi-transparent. It is then settled for some hours, and the lye drawn off. It is then settled

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or fitted by boiling up with water and allowing to settle for some days. This removes excess caustic lye and any insoluble impurities. The contents of the kettle separate into three layers, the soap on top, lye at the bottom and between them a dark colored layer called nigre, containing caustic lye, soap, water and various organic impurities. The lye and nigre are drawn into separate tanks and the soap is pumped to the crutcher, which is a very efficient mixing machine. After being thoroughly crutched, the soap is run into frames, where it stands for some days. It is then cut into cakes, pressed and sold.

Saponification as Carried out in the Laboratory.

A saponification of tallow was carried out as follows:-

The tallow had a saponification number of 195.0; the amount used was 500 gms. of tallow, which would require 97.5 gms. NaOH. This was added in four portions, of 24.4 gms. each, the alkali being added in a lye of 15° Be'.

The additions were as follows:-

Melted tallow	500 gms.
Water	275 cc
Lye, 19% NaOH = 29°Be'	125 cc

The above was boiled into an emulsion before any additions were made.

The next addition was

Water	125 cc
Lye	125 cc

Boiled again, then added

Water	125 cc
Lye	125 cc

Boiled again, then added

Water	125 cc
Lye	125 cc

This completed the theoretical amount of alkali. The soap was completely saponified, and grained by the addition of concentrated salt brine. The soap was then allowed to settle and solidify, over night, and the brine run off. The soap was then boiled up with water, grained and again allowed to cool. After separating this brine, the process was repeated. Finally, the soap was separated from the brine as before, melted, and allowed to cool. The resulting product was pure white and contained but little water. It rapidly dried out, and formed a mass which was granulatedin texture, and crumbled easily. It did not lather freely. Its composition was as follows:-

Moisture	27.4%
Analyses on dry basis:	
Alkali as soap	11.75%
NaCl	8.48%
Unsaponified	.87%
Unsaponifiable	.51%
Fatty anhydride	57.9%
Free acid (as Oleic)	.42%

The next attempt was a saponification of Cottonseed oil.

Stock-- Cottonseed Oil	
Lye	28° Be′
Saponification Number of the fat	185
Amount used	350 grams
Amount lye	64 grams
Amount lye 28° Be′	240 cc.

The saponification was carried out in four steps, using 60 cc of alkali and 240 cc of water in each step. This makes a lye of 14° Be'. The soap was emulsified and boiled as in the case of the tallow. When completely saponified, the soap was grained with about 30 cc of lye at 30° Be', settled, cooled, and the lye drawn off. It was boiled up with water for an hour, and grained with the least possible amount of solid sodium chloride, and again settled and the water run off. This soap was of much better quality than that first made. It was solid, and lathered fairly well. It did not dry out very fast. It analyzed as follows:-

Moisture	35.9%
Dry basis	
Alkali as soap (Na_2O)	10.2%
Unsaponifiable) Unsaponified)	.7%
Free acid (as Oleic)	.35
Fatty anhydride	68.9%

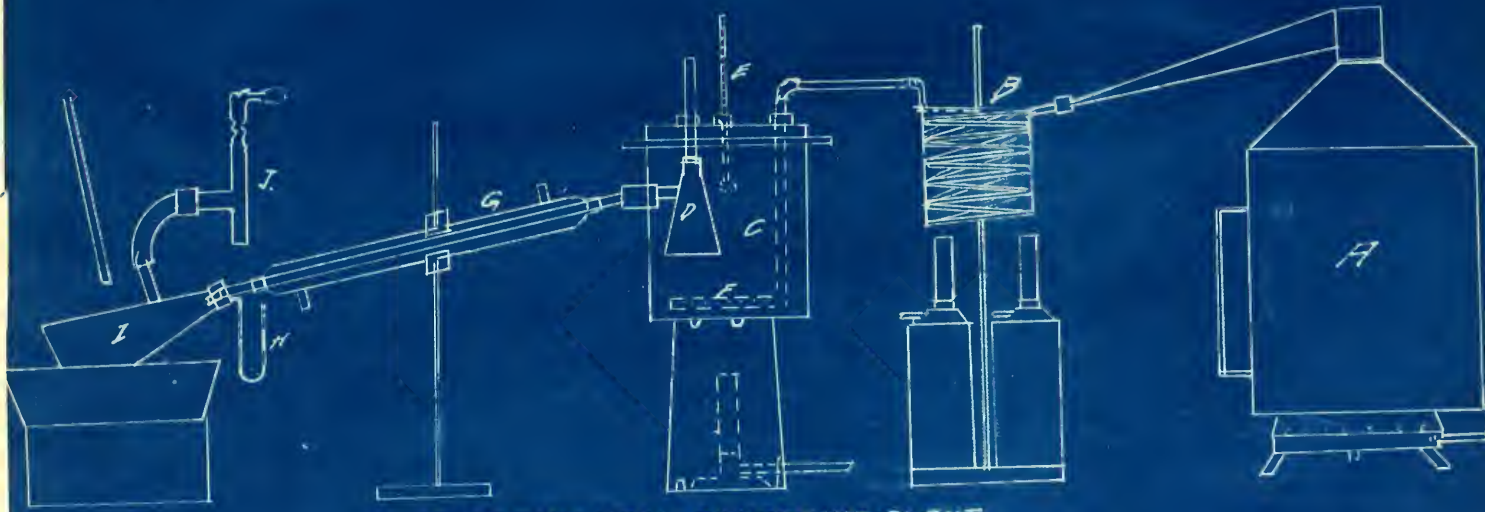
The following methods were followed in the above analyses:-

THE RECOVERY OF GLYCERIN FROM SOAP LYE.

Glycerin is an organic compound, its formula being $C_3H_5(OH)_3$. It is a biproduct that is obtained when fats are saponified with alkali, according to the equation

$$C_3H_5(C_{18}H_{35}O_2)_3 + 3NaOH = C_3H_5(OH)_3 + 3C_{18}H_{35}O_2 \cdot Na$$

The spent lye is drawn from the soap kettles, and sent to the glycerin plant, where it is purified, its alkali neutralized, evaporated, the salt recovered, and the crude product distilled in vacuo with the aid of superheated and expanded steam. As we carried out our tests with very crude apparatus, the results come far from equalling those obtained in practice. The following is a description



EXPERIMENTAL GLYCERINE PLANT

1/8th Size

A. Steam Boiler.
B. Superheating Coil
C. Glycerine Still
D. Air Cooled Receiver
E. Thermometer
F. Open Superheat Steam Pipe.
G. Water Cooled Condenser.
H. Mercury Manometer.
I. Condensed Steam Receiver.
J. Water Suction Pump

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of the process employed and of the results obtained. The spent lye used was obtained from Armour and Company. Its composition was:

Sp.Gr.	1.07
NaOH	.17
Na_2CO_3	.20
Total alkali is NaOH	.31
NaCl	7.31
Glycerin	3.16

The lye contained a large amount of organic matter, both suspended and in solution. The sample taken weighed thirty-one pounds. In order to remove the organic impurities, .5% of alum (36 gms) was added, which precipitated them as an aluminium soap. The lye was then neutralized with the exact calculated amount of sulphuric acid (1.84) 15 cc., and filtered. The lye was then evaporated in an open kettle until most of the salt had crystalized. This was separated by filtration, and the remaining lye treated with 30 cc of a 10% solution of barium chloride. This treatment was made because the half crude glycerin contained considerable organic impurities, which could be precipitated as a barium soap. This soap was filtered off, and the evaporation continued, until crude glycerin was obtained, which analyzed 75.1% glycerin. It was separated as completely as possible from the salt, and distilled with steam as below described.

The apparatus used in this disillation consisted of a steam boiler, A, which supplied ateam which passed through a superheating coil B, heated by two Bunsen burners. The still proper, C, an air condenser D, a Leibig condenser G and receiver I.

The boiler had a capacity of about four gallons. The superheator was a coil of one-eighth inch copper tubing, through which the steem was passed. The still was of spun copper, provided with a tight fitting cover, a place for a

thermometer, and inlet and outlet pipes. The inlet pipe extended to the bottom of the still, and was provided with holes to allow the passage of steam. The outlet was connected to a 500 cc filter flash, which served as an air condenser; the outlet of the filter flask was connected to a long Leibig condenser, which condensed the steam and the glycerin that was not condensed in the air condenser. The condenser lead into a receiver through an air tight joint, this receiver being a 1000 cc filter flask. A filter pump or aspirator served to produce the suction, which was applied at the receiver. A bent U tube filled with mercury served to indicate the vacuum in the system.

Steam was generated in the boiler, and had through the superheater. The still was filled about one-fourth full of crude glycerin, and heat applied with a Bunsen burner. The superheated steam passing through the hot crude glycerin carried some glycerin vapor out of the still. This vapor was partially condensed in the air condenser, which was cool enough to condense the glycerin, but too hot to condense much water. The remaining glycerin condensed with the water and some volatile organic acids in the last receiver. In order to prevent volatile fatty acids from distilling with the glycerin, about 1% of sodium carbonate was added to the glycerin in the still. This combines with the acids, forming non-volatile sodium salts, which remain in the still. These consist chiefly of sodium acetate, which is recovered as such in practice and sold. Considerable amounts of sodium butyrate and sodium caprylate are also formed, but do not find a market. In our first attempt to distill the crude glycerin, we did not heat the still directly, with the result that the steam condensed therein instead of carrying over the glycerin vapors. The glycerin had to be again evaporated before the next attempt was made. Owing to there being too much water in the glycerin, the mixture boiled over into the receiver, while the temperature in the still was only 130° C. The glycerin was again concentrated and filtered, a

little more sodium carbonate added and heated to 110° C. The cover was fastened down, and superheated steam passed again. Great care was taken this time to make all connections very tight, the steam was superheated to a higher degree and a flame again placed under the still. This attempt was successful in securing glycerin, although a considerable amount failed to condense in the air condenser. Glycerin started to come over at 190° C, but it was found that 210° was conducive to greater efficiency. Much higher than this resulted in too great decomposition of glycerin, and even 210° is much too high. For this reason, as great a degree of vacuum as practicable is always employed in practice, thus lowering the temperature to about 320 F (160 C) for the vapors passing off, and 300° F (150°) C for the incoming steam. This keeps the temperature below the point where decomposition products are formed. The best vacuum we could obtain with our suction pump was about three inches of mercury.

The product of this distillation was dark in color, and of strong odor. It contained only 60% glycerin, the remainder being mostly water. It was found that the Leibig condenser became rather warm, and that the condensed sweet water contained much glycerin. This was evaporated and that from the air condenser concentrated in evaporating dishes. In order to condense more glycerine in the air condenser, the copper delivery tube was increased in length from one foot, to three feet. This gave greater air surface and was successful in gaining the desired result. The condenser was changed for one of much greater capacity. We were then able to obtain a vacuum of four inches in the system. More sodium carbonate was then added to the glycerin, and it was redistilled. By paying careful attention to all details, and with the changes in the apparatus just mentioned, nearly colorless glycerin, of about 87% (by analysis) contentration was obtained. Analysis of the sweet water showed only 1.5% glycerin, which is about what is obtained in practice. The distillation was not carried to its

finish, as the last portion contains decomposition products which would contaminate the product. The glycerin in the air receiver was then passed through bone charcoal, the resulting product being absolutely colorless, and with slight odor. It contained 87.2% glycerin. In standing, it turned yellow.

The following are the results obtained:

Sp.Gr.	1.074	Composition of spent lye used.
NaOH	.17	
Na_2CO_3	.30	
Total alkali as NaOH	.51	
NaCl	7.31	
Glycerin	3.16	

Weight of lye used = 501 oz.

Total glycerin present = 440 grams.

Added 0.5% alum = 36 grams

Added 15 cc 1.84 H_2SO_4

30 cc 10% $BaCl_2$ added to half crude containing 35% glycerin.

Free fatty acid test on half crude showed none present.

Crude glycerin 75.1% 438gms.

.751 x 438 = 370 gms. pure glycerin

Total originally present, 440 gms.

Loss 70 gms.

Amount salt present at start 36.62 oz.

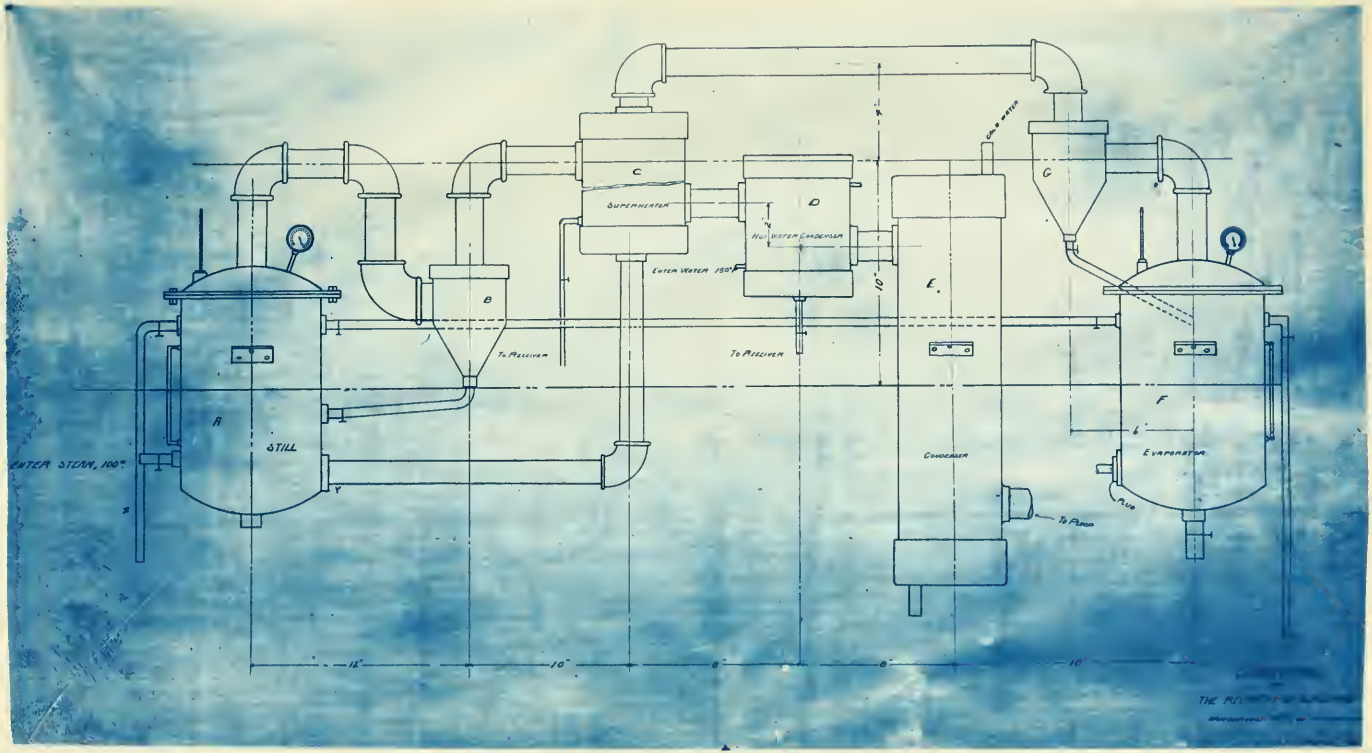
Amount recovered 38.00 oz.

The excess is from the neutralization by H_2SO_4

Amount of glycerin after distillation

120.5 gms. Sp.Gr. 1.187	= 86.8 gms. pure	
40.5 gms. Sp.Gr. 1.10	= 16.2 gms. pure	
21 gms. concentrated	= 21 gms. pure	
	Total 123 gms. pure	

Yield = 123/440 = 28%


A
STILL
B
C
SUPERHEATER
D
HOT WATER CONDENSER
E
CONDENSER
F
EVAPORATOR
To Receiver
To Receiver
To Pump
Enter Water 150°F
ENTER STEAM, 100°
12'
10'
6'
6'
10'

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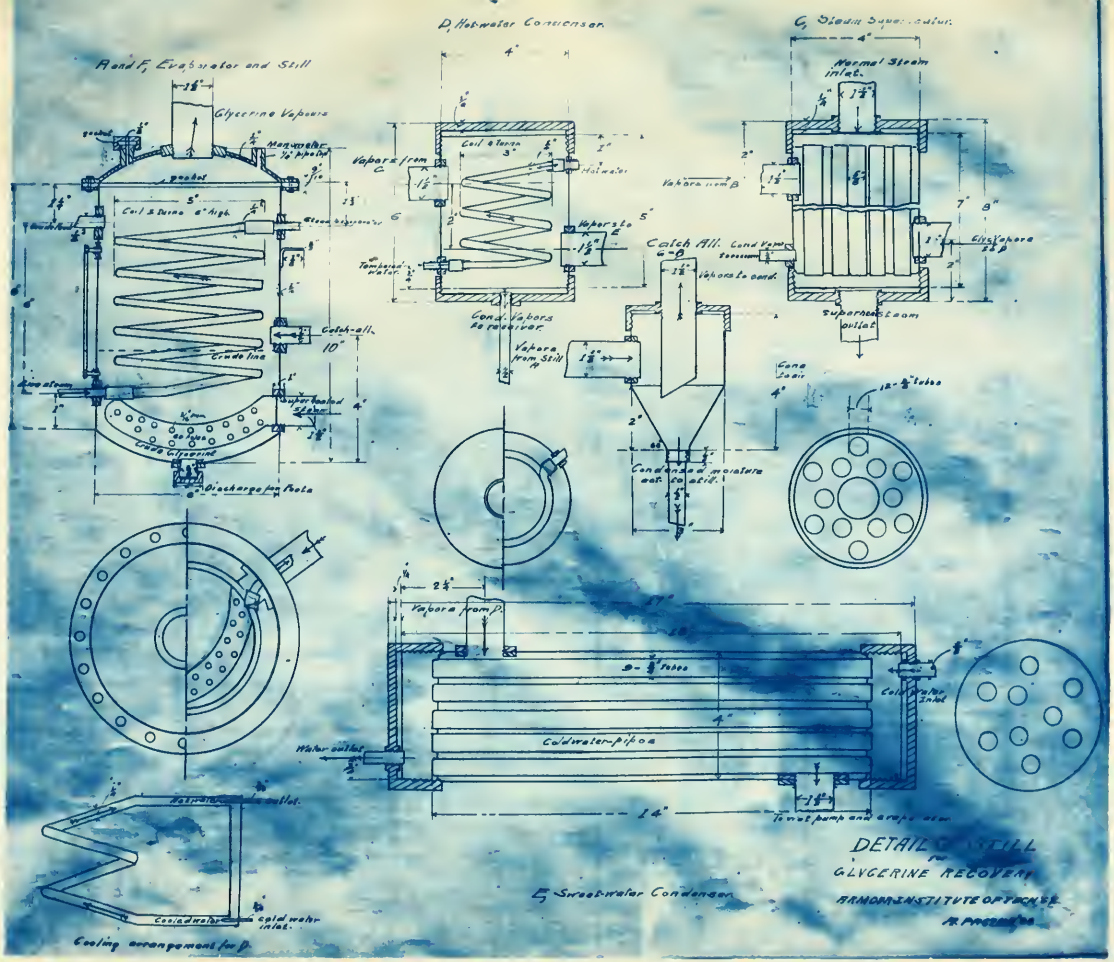
A and F, Evaporator and Still
Glycerine Vapours
Manometer
Coil 2 turns 6" high
Steam to coil
Catch-all
Crude line
Low steam
Discharge for Prots
D, Hot-water Condenser
Coil 6 turns
Hot water
Vapors from C
Vapors to E
Temperature meter
Cond. Vapors to receiver
Vapors from Still A
G, Double Superheater
Normal Steam inlet
Vapors from B
Vapors to cond.
Superheated steam outlet
Catch All. Cond Vapors to cond.
Vapors to cond.
Cone Door
Condensed moisture and solids
Vapors from D
9- ¾" tubes
Cold water inlet
Cold water pipes
Water outlet
Hot water outlet
Cooling arrangement for D
E, Sweet-water Condenser
cold water inlet
To hot pump and evaporator
DETAIL OF STILL for GLYCERINE RECOVERY
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DESIGN OF STILL FOR GLYCERIN.

It can be seen from the poor efficiency and inferior product that was obtained in the laboratory apparatus we used, that many changes must be made before anything approaching heat economy can be realized. It is also necessary, before a concentrated product, containing no organic impurities or decomposition products can be obtained, to have proper condensing devices, and a distillation at a temperature not to exceed 160° C. This is obtained by the use of vacuum about 26 in. mercury being used in practice. If steam directly from the boilers be sent through the glycerin, when the system is under high vacuum, the steam in expanding will absorb a large amount of heat, which it will take from the glycerin in the still, thus preventing its conversion into vapors. Hence it is necessary to expand the steam before it enters the still. Only two systems have been devised which are successful, these being rigidly covered with patents. The "Jobbins and VonBumbech" system expands the steam in suitable coils before sending it into the still. The other system, the "Garrigues" still, is very ingenious in its return to the system of heat that would otherwise be lost. Believing it to be the best, we have chosen that system as the basis of our design. This still is designed to distill about 2000 cc. of glycerin per hour. It consists of the following parts, the letters referring to the assembled drawing:

A Glycerin still

B Catchall

C Superheater

D Hot water condenser

E Sweet water condenser

F Sweet water evaporator

G Catchall.

Crude glycerin, about 75-80% is run through the pipe Z, by means of the vacuum in the still. The level is maintained nearly constant, by running in crude glycerin as the distillation progresses. The superheated and expanded steam passes into the still through the bent pipe Y, which contains numerous small holes on the under side. As this steam passes through the glycerin, it carries with it, from one third to one half its weight of glycerin. Any solid or liquid matter is sent back to the still by being caught in the catchall B. These vapors are heated to about 320° F, or 160° C, this being done by means of the steam coil in the still through which steam at 100 lbs. pressure is sent. As the vapors of glycerin and water pass through the superheater, they meet surfaces that are warmer than the condensing point of water, but so cool that glycerin will condense. This is done by passing the sweet water vapors through in the opposite direction, as here after described. The vapors still contain a large amount of glycerin, so they are passed through condenser D, which contains a coil through which a water at 150° F (65° C) or above is circulated so that no water vapors will condense. In good practice, after passing through these two condensers, the sweet water contains only 1.5% glycerin. This sweet water is condensed in the cold water condenser E, from which it passes through the vacuum pump into a suitable recepticle. The pump must be sufficiently powerful to maintain 24", if possible 26 inches of mercury. It is desirable that not all the glycerin be condensed in the first two condensers, because there is still some volatile fatty acids remaining, which are condensed in the sweet water. The glycerin in the sweet water is recovered by evaporating to a crude glycerin, and redistilling as before. This evaporation is carried out in the sweet water evaporator F, The evaporation is carried on under 24-26 inches of vacuum, and the steam lead through the catchall G to the superheater. Here it meets the hot glycerin vapors from the glycerin still, and is superheated there by. The steam being much cooler

than these vapors, condensers glycerin without condensing water, The superheated steam is then lead into the still, and through the glycerin as before. The steam used to heat the glycerin still is also sent through the steam coil in the sweet water evaporater. Thus it can be seen that the heat efficiency of this apparatus is about the best that can be obtained.

Calculations of heating surfaces, pipes, etc.

These calculations are made on the basis of 665 mm. or 26 in. of vacuum and for a capacity of 6000 cc of sweet water evaporated per hour.

Given 650 mm. vacuum;

Steam at 150° C.

100 liters per hr. per sq. meter of heating

Surfaces can be evaporated.

We will start with an evaporator of following dimensions:-

Diameter	15.24 cm.	= 6"
Height	20.32 cm.	=8"
Cross section	182 sq. cm.	= 28.27 sq. in.
Volume	3700 cc	= 226 cu. in.

Since we desire to evaporate 6000 cc per hour, we will need 6/100 = .06 sq. meters of heating surface = 600 sq. cm.

Use a copper pipe 3/16" = .476 cm. in diameter, and let the pipe be made into a coil 5" (12.7 cm) in diameter and of 10 turns.

The heating surface will be:-

12.7 dia. = 40 cm. in circumference

.476 cm. dia. = 1.5 cm. in circumference,

Each turn has area of 1.5 x 40 = 60 sq.cm.

10 turns have area of 600 sq. cm.

Diameter of Pipes.

The volume delivered will be 6000 cc per hr. or 1 2/3 cc per sec. of liquid. At 665 mm. the steam is expanded to 10000 volumes. Hence, we have 16,600 cc's of water vapor passing through the system. Good practice is to allow a velocity of 15 to 20 meters per second for the steam. Assume 1. 2/3 meters per second = 1660 cm. per second. Then the area of the pipe must be $\frac{16600}{1660}$ = 10 sq. cm. = 1 3/8 inches.

Sweet Water Condenser.

We must condense 6000 cc of water per hour. Heat of steam will be about 600 cal. Then we have 600 x 6000 = 3,600,000 cal. per hour, Allow 18° C rise in temperature of cooling water, We must have

$$\frac{3,600,000}{18} = 200,000 \text{ cc cooling water per hour} = 55.56 \text{ cc. per second.}$$

Allow a velocity of 20 cm. per sec., we must have an area of about 3 sq. cm. to carry this water, Hence, we need 6 tubes each of .5 sq. cm. area = 5/16 in. in diameter.

However, in order to secure a good vacuum, say of 705 mm. or 28" of mercury if we allow a water velocity of 21 cm. per sec. and a steam velocity of 16.6 meters per sec., we will need a surface of 1020 sq. cm., since with the above conditions, a surface of 1.7 sq. meters of copper has been found to condense 100 kilos of steam per hour.

$$\text{Surface} = \frac{\quad}{100} \times 1.7 \times 10000 = 1020 \text{ sq. cm.}$$

To secure this surface, 10 pipes, each .8 cm. dia. = 2.5 cm. circumference, were used. The length was made 40 cm. = 1. in. and the area figures out to be 1020 sq. cm.

In order to secure a good heat efficiency, all exposed parts should be well covered with asbestos. All pipes have been made of the same diameter

(1 3/8 in) as it is not probable that 26" can be obtained unless the fittings be very well made. Also, the velocity of the steam vapors can be increased 50% without injuring the efficiency. In operating this still, it is probable that the vapors will not be heated to more than 180° F. This will not injure the efficiency materially. The glycerin plant at Armour & Company's soap works is of this type, and the temperature in the superheater did not go above 180° F, when 26" vacuum was in use. It is intended that the receivers from the two glycerin condensers be of strong glass such as a filter flask and that they be attached by means of rubber stoppers. In this way the progress of the distillation can be watched.

The references used were

Modern Soaps, Candles and Glycerin by Samborn

Industrial Chemistry by Thorp

Multiple Effect evaporators by Housebrandt

METHODS OF GLYCERIN ANALYSIS.

The following methods of analysis were allowed in this investigation.

Waste Soap Lye. Tests made

Sodium chloride

Total alkali is NaOH

Free alkali NaOH

Combined alkali as Na_2CO_3

Glycerin

Specific Gravity

Total Alkali:

Transfer by means of a pipette to a 500 cc. flask 10 cc of the sample free from soap and foreign matter. Add about 150 cc's of distilled water. Add phenolphthalein as indicator; run in from a burette sufficient half normal sulphuric acid to permanently discharge the pink coloration produced by the indicator; boil to expel all traces of carbonic acid gas (5 min.) and titrate the excess acid used with N/2 caustic soda. To ascertain the percent of total alkali expressed as NaOH, multiply the number of cc's of $N/2H_2SO_4$ absorbed by 0.2

Alkali as Na_2CO_3(Combined alkali), and Free Alkali, as NaOH.

10 cc of the sample, free from foreign matter, is titrated directly with half normal acid, using methyl orange indicator, care being taken not to pass the end point. Phenolphthalein is then added, and the end point again found. Since methyl orange is not sensitive to carbonates, the first titration represents the alkali as NaOH. Both together represent the total alkali, while the difference between the first and second titration gives the amount of alkali as carbonate.

Specific Gravity:-

This was taken at 15° C. with a Westphal balance.

Glycerin, Bichromate Oxidation Method.

In this test, the glycerin is oxidized by the use of potassium bichromate and sulphuric acid, in accordance with the following reaction:

$$3\ C_3H_5(OH)_3 + 7K_2Cr_2O_7 + 28H_2SO_4 = 7K_2SO_4 + 7Cr_2(SO_4)_3 + 80H_2O + 9CO_2$$

Solutions:-

Potassium bichromate.

75 gms. $K_2Cr_3O_7$ and 150 gms H_2SO_4 Sp.Gr. 1.84 per litre.

Ferrous Ammonium Sulphate:-

300 gms. $Fe(NH_4)(SO_4)_2$ and 10 cc H_2SO_4 Sp.Gr. 1.84 per litre.

Solution of Potassium ferricyanide as indicator:-

Standardizations:-

To standardize the ferrous ammonium sulphate, run out 7 to 10 ccs of the bichromate solution into a 250 cc beaker; add 15 cc of H_2SO_4, Sp.Gr. 1.84, and heat for one half hour on the steam bath; dilute to 200 cc and titrate. To standardize the bichromate solution against glycerin, weigh out 0.05 gm. Chemically pure glycerin into a 250 cc beaker, add about 15 cc of bichromate solution, and then slowly 15 cc of H_2SO_4(Sp.Gr. 1.84); heat for one half hour on steam bath and titrate the excess of bichromate with ferrous ammonium sulphate solution. In these titrations, the potassium ferricyanide indicator is used externally on a porcelain tile.

Procedure:-

Weigh out 1.5 - 2.0 gms. of sample into a 25 cc grad. flask, and add 1.5 g moist silver sulphate, weighed roughly. The solution is made up to the mark with distilled water and 6 drops of water are added to displace the volume of the precipitant added. The contents of the flask are then shaken until all the sodium chloride has been precipitated, indicated by the solution being perfectly clear; after which the supernatent liquid is filtered. A portion of the filtrate is

used to rinse the 5 cc pipette, 5 cc are now pipetted into a 250 cc beaker. Bichromate solution, 7 to 10 cc, is now run in, after which 15 cc of (1.84) Sulphuric acid are added. The contents of the beaker (at all times covered wi a watch glass) are heated for one half hour on the steam bath. The solution is then diluted to about 200 ccs. and the excess of bichromate is titrated with ferrous ammonium sulphate solution. The utmost uniformity of procedure must b followed, especially as to the excess of bichromate, and to the degree of diluti

Determination of Sodium Chloride.

Transfer by means of a pipette, 5 cc of sample to a 100 cc beaker and add 50 cc distilled water. Mix thoroughly and transfer 5 cc of the diluted waste l to a 4" porcelain evaporating dish (a casserole is better). Neutralize with dilute nitric acid or sodium carbonate solution, using phenolphthalein as indicator. When neutral add 1 cc of potassium chromate solution as indicator, and titrate to a permanent reddish tinge with N/10 silver nitrate solution.

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